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High surface quality of sintered silicon carbide by chemical mechanical polishing

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Abstract

With the chemical mechanical polishing (CMP) technology the material removal is made with conventional abrasives and chemical dissolution instead of using expensive diamond abrasives. In this investigation, we report on successful CMP polishing of Sintered Silicon Carbide (SSiC) wafer surfaces using concentrated colloidal silica slurries with different pH values. The selection of polishing process parameters (speed of the rotating disk, applied force) are analysed in JMP® (SAS institute) while a design of experiments (DOE) is employed. The optimal wafer surfaces were achieved with colloidal silica CMP under conditions that combine proper parameters (determined by the DOE evaluation) and a high slurry alkalinity (pH > 8.5). Confocal scanning microscopy indicates a significant reduction of roughness and the material removal rate is measured by a touch probe. This process can be combined with dicing and DRIE (Deep Reactive Ion Etching) technology in the tool machining industry for SSiC wafer processing.

Keywords: Chemical mechanical polishing, Sintered Silicon Carbide, Design of Experiment, Surface roughness, Colloidal silica slurry

1. Introduction

A surface processing technology known as the chemical mechanical polishing (CMP) is applied to polish the surface of SSiC samples. SSiC substrates of high surface quality are used for manufacturing of biomedical micro devices, bearings and optics. The material fulfills the needs of optical and tribological applications and it is also implemented in die, mold and tool industry [1-3]. The demands to improve cutting tool technology and the applied materials increase more and more. The SSiC is a high performance ceramic that can be used as an alternative tool material to diamond and cubic crystalline boron nitride (CBN) for processing ferrous materials [4].

SSiC belongs to the advanced ceramics having superior material properties, i.e. high chemical and wear resistance, low density, high hardness and low thermal expansion. Wafer surface preparation of SSiC samples deals with a number of challenges in processing and in modification of surface treatment [5]. The disadvantage of the commercially available samples of SSiC is the roughness of the surface. Smoothness of the substrates is required to apply thinfilm processes such as photolithography. Furthermore the SSiC contains micro pores in contrast to 4H or 6H polycrystalline types of silicon carbide. These pores may affect its mechanical stability. The desired wafer surface qualities such as high uniformity and low surface roughness are mostly ensured by CMP.

Alkaline silica-based slurries are already pinpointed and investigated as a polishing solution to polish SSiC with high surface quality performance [6, 7]. The pH-value of the slurries has a significant influence on the performance of the CMP process to polish SSiC. The surface roughness, uniformity and material removal rate (MRR) is affected [7].

To allow a scientific interpretation of the collected data, a design of experiments (DoE) in combination with statistical evaluation of data obtained from polishing of SSiC is employed. Data is analysed in JMP® (SAS institute) using analysis of variance method (ANOVA) by least-square fit [3], while significant impact parameters are analysed based on the results.

Table 1 Technical properties of NALCO™ polishing slurries

Properties	NALCO™ slurries
Particle Diameter, nm	50-70
Specific Gravity	1.35-1.38
Percent Sodium	0.4
Percent Silica	46-50

2. Experimental procedures

For the planarization of the 100 mm diameter SSiC wafer a rotary polishing machine (P.-Wolters 3R4) is used (Fig. 1), applying a micro-porous polyurethane polishing pad (Dow® IC1000™) and a wafer carrier for the optimal fixation of the wafer. The slurry feeding is carried out manually and the wafer carrier and the disk are rotating in the same direction. In this investigation, the speed of the rotating disk and the applied pressure are varied in the range [40, 60] rpm and [5, 10] kPa, respectively. Three active polishing slurries (NALCO™ 2350, NALCO™ 2354 and NALCO™ 2360) are used with different pH values adjusted by the supplier (NALCO Comp.) to 8.5, 11.0 and 12.0 at 25°C. The most important characteristics of the alkaline slurries are summarized in Table 1.

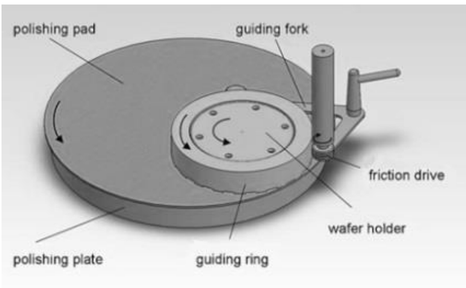

Figure 1. Schematic of the chemical-mechanical planarization machine

2.1. Statistical parameter analysis

In this study three parameters are selected: the pH value of the slurries (pH), the speed of the rotating disk (rpm) and the applied pressure (kPa). All other influencing parameters such as processing time, rotation speed of the carrier, polishing pad etc. are kept constant. Based on knowledge gained in previous tests it is known that these factors would have high influence on the polishing results. Using JMP® software, a fractional factorial design is generated (Table 2) to evaluate the effects of variations in polishing parameters using the results of the confocal scanning microscopy (CSM) and touch probe measurements. In each polishing run a new SSiC wafer is used. To ensure comparability the wafers are lapped and examined subsequently with regard to their surface roughness and wafer thickness.

Table 2 Design of experiment for parameter study

Run #	**Pattern**	**pH**	**speed rpm**	**Pressure kPa**
1	+++	12	60	10
2	+-+	12	40	10
3	++-	12	60	5
4	---	8.5	40	5
5	+--	12	40	5
6	--+	8.5	40	10
7	-++	8.5	60	10
8	-+-	8.5	60	5

4. Results and discussion

DOE has been used for the design of multi-factor experiments. It provides efficient data collection and helps to reduce the workload effectively. It also includes effective problem structuring, comprehensive data analysis that leads to more precise estimation of experimental errors.

By applying DOE, the numbers of experiments are reduced and the experiments are performed according to the design in Table 2, in eight polishing runs. The results obtained are arithmetical mean average (Ra), ten-point mean roughness (Rz) and material removal rate (MRR). All the results are analysed with JMP® by least-square modelling to compare the mean difference and to obtain accurate values of the influence of these three factors.

Table 3 Parameter estimates on Ra/Rz

Term	**Estimate**	**Standard error**	***t* ratio**	**Prob >|*t*|***
pH (8.5-12)	0.007/0.04	0.036/0.19	0.2/0.2	0.87/0.87
speed (40-60)	0.025/0.14	0.032/0.17	0.8/0.82	0.57/0.56
pressure (5-10)	0.003/0.001	0.032/0.17	0.1/0.01	0.93/0.99

*Prob |*t*| stands for the probability of observing |*t*| value which is greater than *t* ratio. It is obtained from the *t* distribution table. [Prob >|*t*|] of 0.05 is the value to define statistical significance.

4.1. Surface roughness Ra and Rz

The Ra and Rz measurement data for each run is fed in JMP® software and analysed using ANOVA method by least-square fit (Table 3). Fitting results reveal that Prob > |t| is greater than 0.05 for pH, rotation speed and pressure. It demonstrates that no significant differences are observed when changing the above factors within the tested ranges. Prediction profile (Fig. 2) shows that Ra and Rz values would lead to an improvement within $0.0 \leq Ra \leq 0.10$ and $0.04 \leq Rz \leq 0.55$ respectively, if parameters were changed within their testing range.

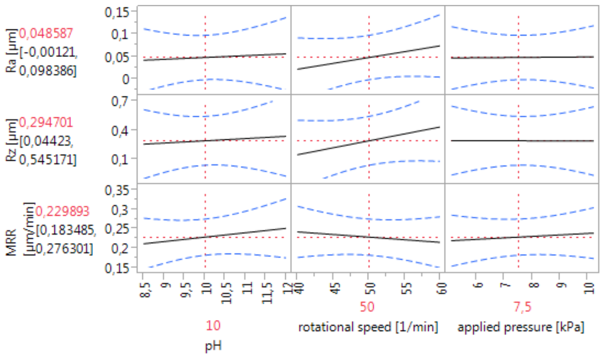

Figure 2. Prediction profiles of the parameters pH, rotational speed and applied pressure on 95% confidence interval

4.2. Material removal rate

The material removal rates are also analysed using the ANOVA method by least–square fit. The fitting result reveals that Prob >|t| is greater than 0.05 for pH, rotation speed and pressure, but the product of rotation speed and applied pressure shows significant differences (Prob ≤|t| is less than 0.05). When analyses are performed within the above control limits, it will give at least 81.9% assurance that the predicted MRR will be achieved.

Table 4 Parameter estimates on MRR

Term	**Estimate**	**Standard error**	**t ratio**	**Prob >|t|**
pH (8.5-12)	0.019	0.017	0.19	0.32
Speed (40-60)	-0.010	0.014	-0.70	0.53
pressure (5-10)	0.009	0.014	0.67	0.55
Speed* applied pressure	0.048	0.014	3.35	**0.044**

5. Conclusion

A parameter analysis for this chemical and mechanical surface polishing is successfully completed through DOE, and data are analysed by JMP® (SAS Institute). Control limits for processing parameters are set in a reasonable range, and prediction profiles for the influence parameters are determined. It is demonstrated that DOE with JMP® software can result in efficient acquisition of data, comprehensive data analysis and precise conclusions for the CMP process. The combination of DOE and JMP® software provides a tool for the systematic analysis of multi-factorial design for high surface quality of sintered silicon carbide by chemical mechanical polishing.

References

[1] Zorman C A 2009 Silicon Carbide as a Material for Biomedical Microsystems EDA Publishing Association

[2] Vanko G, Hudek P, Zehetner J, Dzuba J, Choleva P, Kutiš, Vallo M, Ryger I and Lalinsky T 2013 Bulk micromachining of SiC substrate for MEMS sensor applications, Microelec. Eng. **110** 260-64

[3] Klocke F and Zunke R 2009 Removal mechanisms in polishing of silicon based advanced ceramics CIRP Annals - Manufacturing Technology **58** 491-94

[4] Choyke W J, D'Urso B, Yan F and Devaty R P 2010 Ultra-Precision Machining of Stainless Steel and Nickel with Single Crystal 4H and 6H Boule SiC Mat. Sci. For. **645-648** 853-56

[5] Powell J A 1997 Chemomechanical Polishing of Silicon Carbide, J. of The Electrochem. Soc. **144** Issue 6 161-63

[6] Li Y Zorman 2008 Microelectronic applications of chemical mechanical planarization, John Wiley & Sons Inc.

[7] Rittinger J, Cvetkovic S and Rissing L 2012 Slurry Comparison for Chemical-mechanical Polishing (CMP) of Silicon Carbide (SiC), 12th euspen int. conf. Stockholm